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Methyl 5-iodo-2-methoxybenzoate

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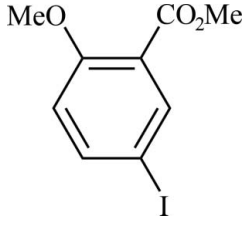
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.033; wR factor = 0.079; data-to-parameter ratio = 17.2.

In the title compound, $C_9H_9IO_3$, the molecules are close to planar [maximum deviation from benzene ring plane = 0.229 (5) Å for the methyl carboxylate C atom] with the methyl groups oriented away from each other. In the crystal, molecules form stacked layers parallel to the *ab* plane, where every layer has either the iodine or methoxy/methyl carboxylate substituents pointing towards each other in an alternating fashion.

Related literature

For the synthesis, see Wang *et al.* (2009).



Experimental

Crystal data

$C_9H_9IO_3$ M_r = 292.06
Monoclinic, $P2_1/n$
a = 4.3378 (7) Å
b = 7.0690 (11) Å
c = 33.120 (5) Å
β = 92.727 (2)°
V = 1014.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.13 mm^{-1}
T = 293 K
0.35 × 0.20 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.407, T_{max} = 0.788
7578 measured reflections
2064 independent reflections
1971 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.079
S = 1.17
2064 reflections
120 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 e Å^{-3}

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) implemented in *WinGX* (Farrugia, 2012); molecular graphics: *DIAMOND* (Brandenburg, 2004) and *ChemBioDraw Ultra* (CambridgeSoft, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: LR2123).

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supplementary materials

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1. Introduction

2. Experimental

2.1. Synthesis and crystallization

The title compound was synthesized by the method used by Wang *et al.* (2009), only differing slightly in the reaction time which was increased from 30 to 60 minutes. The ^{1}H NMR spectrum of the title compound is in good agreement with what was reported by Wang *et al.* (2009). The title compound was dissolved in $CDCl_3$ for NMR-analysis, and slow evaporation of the solvent yielded single crystals suitable for X-ray diffraction.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The structure was refined by full-matrix least squares using *SHELXL97* (Sheldrick, 2008) as implemented in the *WinGX* suite (Farrugia, 2012). H-atoms were positioned geometrically at distances of 0.93 (CH) and 0.96 Å (CH_3) and refined using a riding model with U_{iso} (H)=1.2 U_{eq} (CH) and U_{iso} (H)=1.5 U_{eq} (CH_3).

3. Results and discussion

The title compound is an intermediate in the synthesis of 4,4′-dimethoxy-3,3′-biphenyldicarboxylic acid, a novel organic linker for use in MOFs (Metal-Organic Frameworks). The title compound is a known intermediate from the literature (Wang *et al.*, 2009), but the crystal structure has not been reported so far.

The structure of the title compound, $C_9H_9IO_3$, has a monoclinic $P2_1/c$ symmetry. The asymmetric unit equals one molecule of the compound, with the full content of the unit cell generated by symmetry operations. The molecule has a planar motif where the methyl groups are oriented away from each other to accommodate the sterical demands of these groups. To further increase the distance between the methyl groups, an alternative configuration of the molecule could theoretically be achieved by rotating the methyl carboxylate group 180° around the C1–C7 bond. This however appears not to be an energetically favourable configuration in the solid state. The asymmetric units are packed to form layers parallel to the *C* plane, which results in a layered structure where every other layer has either an iodine or a methoxy/methyl carboxylate interface.

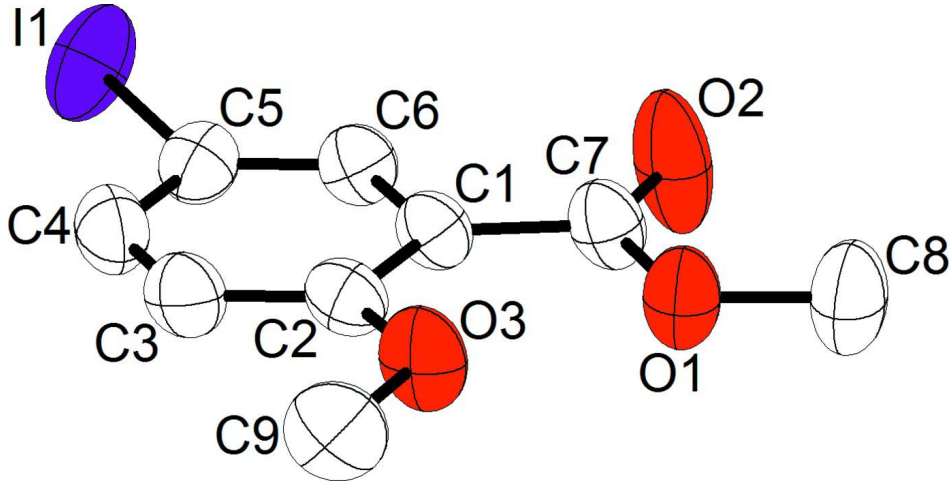

Figure 1

One molecular unit of the title compound with 50% probability displacement ellipsoids. Hydrogen atoms are omitted for clarity.

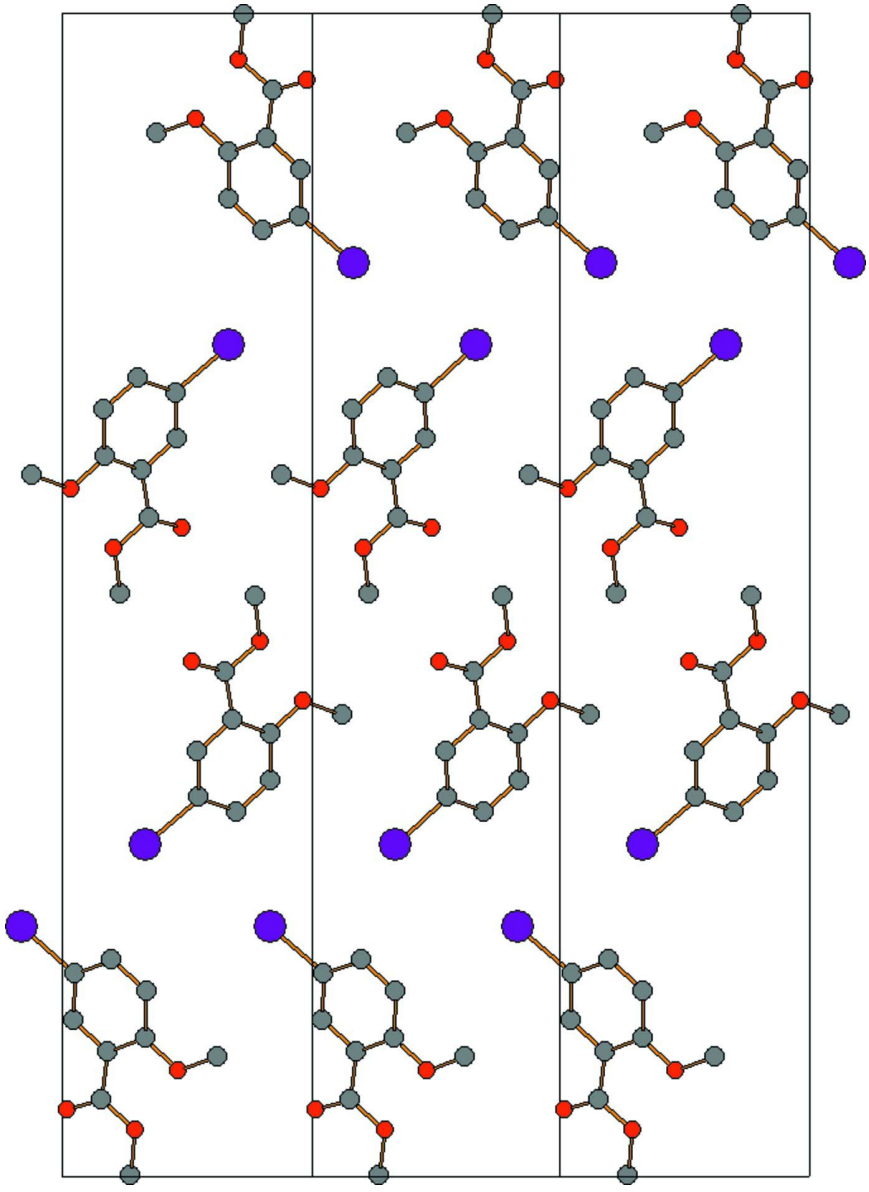

Figure 2

Packing diagram of the title compound viewed along the *a* axis. Hydrogen atoms are omitted for clarity.

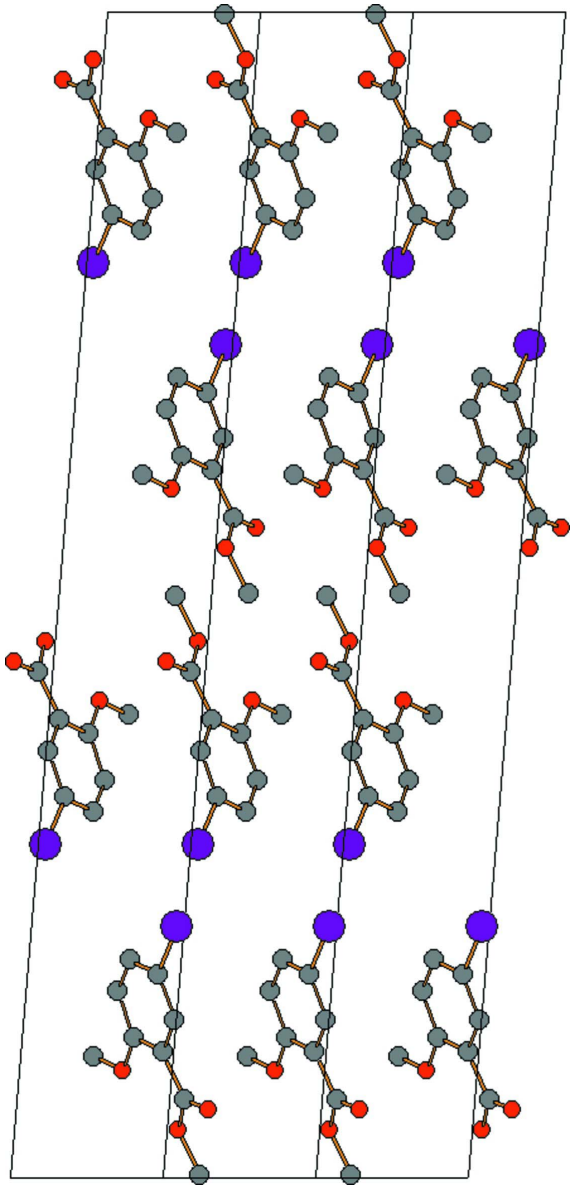

Figure 3

Packing diagram of the title compound viewed along the *b* axis. Hydrogen atoms are omitted for clarity.

Methyl 5-iodo-2-methoxybenzoate

Crystal data

$C_9H_9IO_3$
$M_r = 292.06$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.3378$ (7) Å
$b = 7.0690$ (11) Å
$c = 33.120$ (5) Å
$\beta = 92.727$ (2)°
$V = 1014.4$ (3) Å^3
$Z = 4$

$F(000) = 560$
$D_x = 1.912$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5646 reflections
$\theta = 2.5$–28.8°
$\mu = 3.13$ mm^{-1}
$T = 293$ K
Plate, colourless
0.35 × 0.20 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.407, T_{max} = 0.788
7578 measured reflections
2064 independent reflections
1971 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 26.4°, θ_{min} = 2.5°
h = −5→5
k = −8→8
l = −41→41

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.079
S = 1.17
2064 reflections
120 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0267P)^2 + 1.5075P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 e Å^{-3}

Special details

Experimental. The recrystallization was performed in deuterated solvent, $CDCl_3$.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C8	1.2399 (13)	0.2713 (7)	0.00134 (13)	0.0750 (13)
H8C	1.1673	0.1559	−0.0110	0.112*
H8B	1.4608	0.2677	0.0048	0.112*
H8A	1.1796	0.3764	−0.0156	0.112*
C7	1.1645 (9)	0.1539 (5)	0.06620 (11)	0.0513 (8)
C1	1.0379 (8)	0.1809 (5)	0.10720 (10)	0.0432 (7)
C2	0.8498 (8)	0.3314 (5)	0.11933 (11)	0.0467 (8)
C3	0.7587 (9)	0.3356 (5)	0.15924 (12)	0.0541 (9)
H3	0.6357	0.4345	0.1676	0.065*
C4	0.8469 (9)	0.1966 (6)	0.18637 (12)	0.0563 (9)
H4	0.7844	0.2022	0.2128	0.068*
C9	0.5840 (11)	0.6212 (6)	0.10360 (16)	0.0719 (13)
H9C	0.5652	0.7115	0.0820	0.108*
H9B	0.6821	0.6798	0.1270	0.108*
H9A	0.3825	0.5775	0.1100	0.108*
C5	1.0295 (8)	0.0478 (5)	0.17421 (11)	0.0486 (8)

C6	1.1226 (8)	0.0425 (5)	0.13512 (10)	0.0450 (7)
H6	1.2462	−0.0571	0.1272	0.054*
O1	1.1074 (7)	0.2925 (4)	0.04032 (8)	0.0614 (7)
O3	0.7663 (7)	0.4639 (4)	0.09139 (9)	0.0652 (8)
O2	1.3145 (10)	0.0189 (5)	0.05808 (9)	0.0984 (14)
I1	1.16959 (7)	−0.16619 (5)	0.215055 (9)	0.07304 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C8	0.101 (4)	0.073 (3)	0.053 (2)	0.023 (3)	0.022 (2)	0.014 (2)
C7	0.065 (2)	0.0454 (18)	0.0436 (18)	0.0166 (17)	0.0017 (16)	−0.0022 (15)
C1	0.0462 (17)	0.0391 (16)	0.0441 (17)	0.0076 (14)	0.0004 (14)	−0.0047 (13)
C2	0.0466 (18)	0.0389 (17)	0.055 (2)	0.0056 (14)	0.0007 (15)	−0.0076 (15)
C3	0.050 (2)	0.049 (2)	0.064 (2)	0.0077 (16)	0.0127 (17)	−0.0122 (17)
C4	0.054 (2)	0.067 (2)	0.049 (2)	0.0002 (18)	0.0088 (16)	−0.0088 (18)
C9	0.075 (3)	0.043 (2)	0.098 (3)	0.026 (2)	0.016 (2)	−0.002 (2)
C5	0.0412 (17)	0.055 (2)	0.0495 (19)	−0.0002 (15)	0.0004 (14)	0.0047 (16)
C6	0.0439 (17)	0.0421 (17)	0.0488 (18)	0.0071 (14)	0.0010 (14)	−0.0018 (14)
O1	0.080 (2)	0.0538 (15)	0.0515 (15)	0.0239 (14)	0.0157 (13)	0.0088 (12)
O3	0.0808 (19)	0.0493 (15)	0.0664 (17)	0.0320 (14)	0.0115 (14)	0.0011 (13)
O2	0.166 (4)	0.076 (2)	0.0566 (18)	0.074 (2)	0.032 (2)	0.0091 (16)
I1	0.05978 (19)	0.0988 (3)	0.06086 (19)	0.01134 (15)	0.00665 (13)	0.03178 (15)

Geometric parameters (Å, º)

C8—O1	1.446 (5)	C3—C4	1.373 (6)
C8—H8C	0.9600	C3—H3	0.9300
C8—H8B	0.9600	C4—C5	1.388 (5)
C8—H8A	0.9600	C4—H4	0.9300
C7—O2	1.193 (4)	C9—O3	1.434 (4)
C7—O1	1.318 (4)	C9—H9C	0.9600
C7—C1	1.501 (5)	C9—H9B	0.9600
C1—C6	1.384 (5)	C9—H9A	0.9600
C1—C2	1.411 (4)	C5—C6	1.375 (5)
C2—O3	1.354 (4)	C5—I1	2.100 (4)
C2—C3	1.398 (5)	C6—H6	0.9300
O1—C8—H8C	109.5	C3—C4—C5	119.8 (4)
O1—C8—H8B	109.5	C3—C4—H4	120.1
H8C—C8—H8B	109.5	C5—C4—H4	120.1
O1—C8—H8A	109.5	O3—C9—H9C	109.5
H8C—C8—H8A	109.5	O3—C9—H9B	109.5
H8B—C8—H8A	109.5	H9C—C9—H9B	109.5
O2—C7—O1	122.4 (3)	O3—C9—H9A	109.5
O2—C7—C1	122.2 (3)	H9C—C9—H9A	109.5
O1—C7—C1	115.3 (3)	H9B—C9—H9A	109.5
C6—C1—C2	118.8 (3)	C6—C5—C4	119.4 (3)
C6—C1—C7	114.7 (3)	C6—C5—I1	119.9 (3)
C2—C1—C7	126.5 (3)	C4—C5—I1	120.7 (3)

O3—C2—C3	123.6 (3)	C5—C6—C1	122.0 (3)
O3—C2—C1	117.8 (3)	C5—C6—H6	119.0
C3—C2—C1	118.6 (3)	C1—C6—H6	119.0
C4—C3—C2	121.4 (3)	C7—O1—C8	115.7 (3)
C4—C3—H3	119.3	C2—O3—C9	118.5 (3)
C2—C3—H3	119.3		

O2—C7—C1—C6	3.2 (6)	C3—C4—C5—C6	0.7 (6)
O1—C7—C1—C6	−174.6 (3)	C3—C4—C5—I1	179.7 (3)
O2—C7—C1—C2	−177.6 (4)	C4—C5—C6—C1	−0.5 (5)
O1—C7—C1—C2	4.6 (6)	I1—C5—C6—C1	−179.6 (3)
C6—C1—C2—O3	−179.1 (3)	C2—C1—C6—C5	−0.2 (5)
C7—C1—C2—O3	1.7 (6)	C7—C1—C6—C5	179.1 (3)
C6—C1—C2—C3	0.6 (5)	O2—C7—O1—C8	−0.3 (7)
C7—C1—C2—C3	−178.6 (4)	C1—C7—O1—C8	177.4 (4)
O3—C2—C3—C4	179.3 (4)	C3—C2—O3—C9	2.4 (6)
C1—C2—C3—C4	−0.4 (6)	C1—C2—O3—C9	−177.9 (4)
C2—C3—C4—C5	−0.2 (6)		